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E. Phillip Muntz		
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)		
University of Southern California Department of Aerospace Engineering Los Angeles, CA 90089-1191		
8. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)		
Dr. Len Sakell AFOSR/NA 110 Duncan Ave., Suite B115 Bolling AFB, D.C. 20332-0001		
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13. ABSTRACT (Maximum 200 words)	

The study of nonequilibrium flow phenomena using a hypersonic flow of iodine vapor was originated under an AFOSR URI grant. At the end of that grant the major components of a large iodine vapor tunnel were available and assembled. There were insufficient funds to provide a satisfactory iodine resistant coating for the interior of the tunnel. Several inexpensive coatings were investigated but were not satisfactory. The decision was made at that time to validate the cryogenic pumping performance of the iodine hypersonic tunnel by using CO₂. In parallel a search was initiated for the approximately twenty-five thousand dollars in equipment money required to coat the interior of the tunnel to permit operation with iodine. The report of the work appears here. The pumping performance of the facility is very satisfactory and exceeds the design requirements. As it turns out the University of Southern California, based on the promising pumping results, has now been able to provide sufficient funds to complete the tunnel coating so that by early 1997 the facility will be able to operate on iodine. The University is also at present supporting the work of a student to complete the facility.

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Final Technical Report
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06/01/93 - 05/31/96
on
Non-Equilibrium Flow Experiments in the U.S.C. Iodine Flow Facility
(Grant #49620-93-1-0390)

E. Phillip Muntz, Principal Investigator
Stephen E. Vargo, Graduate Student
Department of Aerospace Engineering
University of Southern California
Los Angeles, CA 90089-1191

Background

Iodine is an attractive gas for the detailed experimental study of nonequilibrium, chemically reacting hypersonic flows. In terms of dissociation, a stagnation temperature of 1,500 K for iodine is equivalent to about 4,000 K for oxygen or 6,000 K for nitrogen. An iodine hypersonic flow facility in which detailed measurements of internal state populations are possible, can be used for the validation of flow prediction techniques when nonequilibrium chemistry is important. A pilot scale hypersonic wind tunnel has been successfully designed and tested. This facility had run times of the order 30 minutes with recirculating times of about 2 hours. The hypersonic flow of iodine vapor was generated using a sonic orifice. Stagnation conditions for this facility were a stagnation pressure(p_o) and temperature(T_o) of 100 Torr and 1,000 K, respectively. A full scale hypersonic facility, based on the results obtained from the pilot scale facility, is currently undergoing tests of its vacuum and cryogenic pumping capabilities. Design flow periods are up to 30 minutes with a stagnation temperature of 1,500 K and stagnation pressure of 10 atm. Relatively long flow times are desirable to permit the detailed probing of the many energy levels that are populated in high energy flows. Accurate measurements, using transient laser induced fluorescence or other techniques¹, of the population distributions for the large number of significantly populated levels typical of a dissociating gas, will permit meaningful tests of the basic assumptions about local thermodynamic equilibrium that are made in most predictions involving finite rate chemistry.

Introduction

A schematic of the full scale hypersonic facility is shown in figure 1. It is aligned vertically and operates intermittently employing a closed cycle. The operating procedure for the full scale facility is basically identical to the earlier pilot scale apparatus. During the run phase the boiler (fig. 1) is maintained at a temperature that provides the desired stagnation pressure. The saturated iodine vapor from the boiler flows to the stagnation chamber, where it is superheated to the stagnation temperature. The high temperature iodine in the stagnation chamber expands through a hypersonic nozzle which exhausts into a vacuum chamber. In the pilot scale facility, a sonic orifice was used to expand the iodine flow instead of a nozzle. Pumping of the working gas during the run is achieved by condensing the iodine on cryogenic panels that are located downstream of the nozzle's exit. The second phase of operation consists of recycling the iodine from the cryogenic panels back to the boiler. This recirculation is achieved by heating the cryogenic panels and the facility's vacuum envelope and cooling the walls of the boiler. When this occurs, the iodine

sublimates and is driven to the boiler by the vapor pressure differential that develops due to the temperature differences within the facility.

Since iodine is a corrosive and toxic gas, much attention was paid to the materials selection and facility design to ensure a reasonable facility lifetime as well as providing for personal safety precautions. When inhaled, iodine may cause eye, nose, throat, and respiratory tract irritation. If exposure is repeated, bronchitis, skin rashes, and loss of appetite and sleep may result. The maximum accepted concentration for continuous exposure is 0.1 ppm, which corresponds to 1 mg/m^3 at standard atmospheric conditions. Reported lethal doses lie between a few tenths of a gram to more than 20 g whole body burden. In the full scale facility, the quantities of iodine that are used will be of the order tens of kilograms at pressures above atmospheric, it is therefore absolutely necessary that potential leaks be contained by the facility's design.

In order to attain design experimental conditions in the full scale facility, the ability of the facility to maintain a high vacuum and the capability of the cryogenic panels to pump large gas loads needed to be determined. The configuration of the pumping system (mechanical, diffusion, cryogenic) is outlined in figure 2. During the recycling phase of the iodine, the mechanical and diffusion pumps are isolated from the chamber (i.e. a gate valve is closed to prevent exposure of the pumps to iodine during recycling); therefore, it is critical that the leak rate of the chamber be reduced to a minimum. In order to validate the pumping capability of the cryogenic panels, tests using equivalent loads of carbon dioxide gas were used to simulate the design volume flow rate of iodine gas through the supersonic nozzle. The cryogenic pump needs to be able to maintain a low pressure downstream of the nozzle exit when a flow of gas exists. In summary, the initial testing of the newly constructed full scale facility consisted of evaluating the no flow vacuum performance with the diffusion and mechanical pumps and determining the pumping capability of the cryogenic panels. A further consideration was that of the vacuum and pumping integrity of the facility before coating the facility walls with a corrosion proof material.

No Flow Vacuum Performance

A diffusion and mechanical pump provide the ultimate pump down pressure that is desired for experimental conditions. A roughing/backing pump with a pumping speed of 150 CFM is used to bring the facility from atmosphere down to a pressure of 20 mTorr. The blank-off pressure of the mechanical pump is 10 mTorr. Once the facility is in the range of 50 to 70 mTorr, a 10-inch diffusion pump delivering a pumping speed of 5,000 L/s is started. The blank-off pressure of the diffusion pump without the use of a cold trap is 1×10^{-7} Torr.

Leak testing of the full scale facility was completed in two stages (figure 2). The first stage consisted of vacuum testing only the cryogenic pumping section. The diffusion pump and mechanical pump were allowed to pump down this section to verify that no significant leaks were present and to condition the interior of the chamber in order to reduce outgassing of the stainless steel walls. For pressure measurements in the facility, two ion and thermocouple gauge pairs were used. A pair was placed on the pump down line elbow next to the diffusion pump and the other was located on a sealing flange placed at the top of the pumped section. These two locations allow one to accurately read the pressure of the diffusion pump and of the pumped section. A pressure of 8×10^{-6} Torr was measured on the top flange and 2×10^{-6} Torr on the elbow for the cryogenic section with using only the diffusion and mechanical pumps. The second stage of leak test-

ing consisted of installing the test section (fig. 2) above the cryogenic section and performing the same vacuum checks. With the test section installed, a pressure of 1.6×10^{-5} Torr on the top flange and 3.7×10^{-6} Torr on the elbow were measured. The addition of the test section and its resultant pressure measurements are a good indication that the facility is vacuum tight and has no major leaks. The entire facility was also leak tested with a helium mass spectrometer leak detector and found to have no detectable leaks.

Pumping Capability of the Cryogenic Panels

The condensation pump is designed for the particular application to the full scale hypersonic facility. When the facility is in the run phase of operation, the supersonic nozzle exit static pressure will be about 100 mTorr. In order to control the condensation pumping at these rather high pressures the condensing surfaces have been arranged as shown in figure 3 and 4. Stainless steel panels are mounted vertically in a star pattern centered about a perforated tube. Two different configurations for the Teflon® sheets within the cryogenic section were tested. Figure 3 outlines the first configuration option and figure 4 outlines the other. In figure 3, the cryogenic panels are separated from the pressure required for balancing the nozzle flow by a cylindrical surface of Teflon containing long vertical slits about 7 mm wide. The slits are positioned at the midpoint of the outer boundary of the wedge shaped sections forming the condensing surfaces. Between the slotted cylinder and the pump's outer vacuum boundary the pressure will be close to the nozzle exit pressure. The vertical slots form continuum two-dimensional jets that expand freely into the wedge shaped spaces. In these spaces the static pressure is less than 1 mTorr. These underexpanded jets, which have associated shock systems located 30 cm downstream of the slits, will impinge directly on the cryogenic surfaces, both enhancing the efficiency of the surfaces and distributing the condensate in a more uniform manner. For thermally isolating the cryogenic surfaces from the walls of the chamber, a cylindrical Teflon surface is placed 20 cm from the slit surfaces. The second configuration of Teflon sheets outlined in figure 4 was selected in order to validate the necessity of an inner Teflon wrap in the facility.

No Flow Vacuum Performance

Initial testing of the panels was performed in order to evaluate the ultimate pressure attainable at no flow conditions. Upon reaching the lowest attainable pressures available with the diffusion and mechanical pumps, the panels were filled with liquid nitrogen. As the panels began to cool, cryopumping began within the facility. The lowest pressure reached with the test section was 5.7×10^{-7} Torr on the top flange and 1.8×10^{-6} Torr on the elbow. Without the test section installed, a pressure of 3×10^{-7} Torr on the elbow and 4.8×10^{-8} Torr on the top flange were obtained. These low pressure measurements are another indication that the facility is vacuum tight.

Design Conditions

The full scale facility is designed to operate on 50 kg of pure iodine, with a stagnation temperature of 1,500 K and pressure of 10 atm. The supersonic nozzle that will be used has a throat radius of 1.778 mm and an exit radius of 10.16 cm. With a downstream pressure of 100 mTorr the corresponding exit Mach number is about 9. From the facility's stagnation conditions and the

nozzle's geometry it is possible to calculate the mass flow rate of iodine gas from

$$\dot{m} = \pi P_o r_t^2 \sqrt{\frac{\gamma m}{k T_o}} \left(\frac{2}{\gamma + 1} \right)^{\frac{\gamma + 1}{2(\gamma - 1)}} \quad (1)$$

with P_o , T_o , r_t , γ , m , and k being the stagnation pressure, stagnation temperature, radius of the nozzle throat, ratio of specific heats, mass of the iodine molecule and Boltzmann's constant. In this facility, the iodine molecules are assumed to be rotationally and vibrationally active, thus a γ value of 9/7 is selected. With the appropriate values for the full-scale facility, the mass flow rate of iodine is 30.2 g/s.

Carbon Dioxide Gas Load Results

In order to simulate the gas load conditions imposed on the cryogenic panels during an actual run a flow of carbon dioxide gas through a free-jet is chosen. This allows one to verify the pumping capability of the cryogenic surfaces without exposing them to the corrosive effects of iodine. Since the simulation uses carbon dioxide instead of iodine, the iodine mass flow rate is converted to a carbon dioxide mass flow rate from the number flow of CO_2 molecules being equivalent to the number flow of I_2 molecules, then

$$\dot{m}_{\text{CO}_2} = \dot{m}_{\text{I}_2} \left(\frac{MW_{\text{CO}_2}}{MW_{\text{I}_2}} \right) \quad (2)$$

with MW representing the molecular weight of the gas. The carbon dioxide mass flow rate required for a simulation of 30.2 g/s of I_2 is about 5.2 g/s. In the actual testing of the cryogenic panels, a volume flow rate of carbon dioxide gas is measured with a flowmeter. The volume flow measurement is converted to a mass flow by knowing the pressure and temperature of the gas flow in the flowmeter. For the carbon dioxide runs, both Teflon configurations as shown in figures 3 and 4 were tested. Figure 5 outlines the results of the carbon dioxide simulations for the facility without the test section installed. Note that the mass flows have been converted to equivalent iodine flow rates. As expected, increasing the flow rates of CO_2 increased the pressures measured in the cryogenic section. Recall that the full scale facility is designed such that the downstream nozzle exit pressure needs to be about 100 mTorr. Both simulations verify that the cryogenic panels would handle the designed gas load of iodine. The second Teflon configuration appears to pump more efficiently than the first configuration. At the designed iodine flow rate of 30 g/s, the second configuration maintains a tank pressure of 6 mTorr whereas the first configuration maintains a pressure of 35 mTorr. The full-scale facility should adopt the second configuration within the cryogenic section since this layout pumps higher flow rates of gas at lower tank pressures in comparison with the first configuration. Also, the second configuration reduces the materials present within the corrosive environment. This configuration will not only cost less to prepare for the corrosive environment but is a much simpler and easier section to assemble. A test of the second configuration under gas load with the gate valve opened was also performed with no pressures detected on the elbow's thermocouple gauge. This is a good indication that the panels are pumping most of the gas flow present in the tank. During this test the ion gauge on the elbow measured between 3 and 4×10^{-4} Torr while pumping gas. This is a good indication that the gate valve may even remain open while pumping the iodine gas load.

Conclusions

Pumping tests of two different radiation shield configurations for the Iodine Hypersonic Wind Tunnel have been conducted. Both configurations meet the required test section pressure requirements (less than 100 mTorr) at the designed mass flow. The single Teflon radiation shield will be used in the facility since it is simpler and easier to implement when requirements for the protective coating of the facilities pumping surfaces are considered.

Acknowledgment

This work was supported by the United States Air Force Office of Scientific Research as part of the Augmentation Awards for Science and Engineering Research Training program. The author would like to thank Mr. Thane DeWitt for his expertise in vacuum and structural welding performed on the full scale facility. Along with Mr. DeWitt, Mr. Mark Trojanowski and Dr. Andrew Ketsdever also added valuable comments and suggestions to the construction of this facility, without these individuals, this project would have not been possible.

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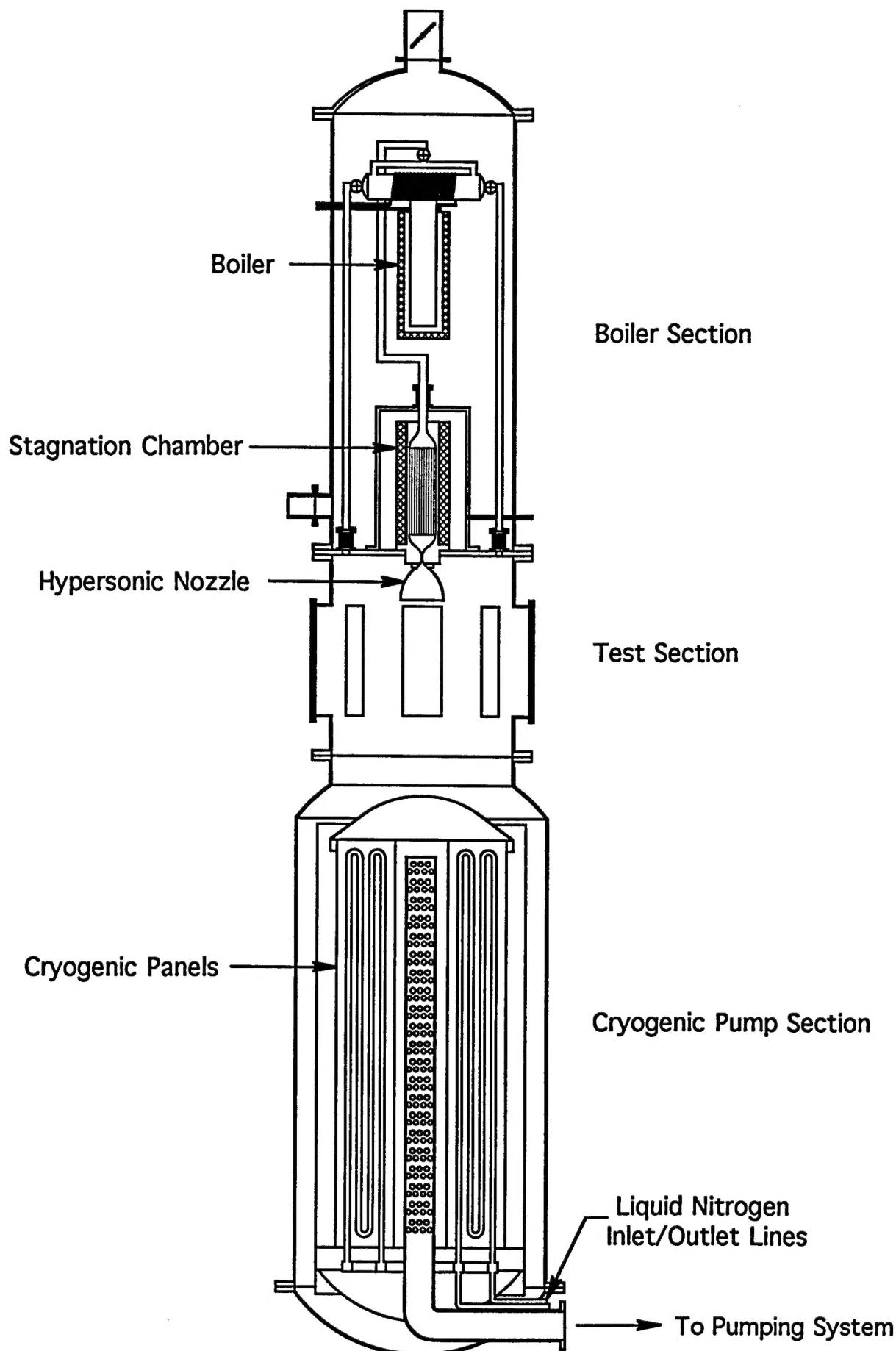


Figure 1: Schematic of the Full Scale U.S.C. Iodine Hypersonic Wind Tunnel.

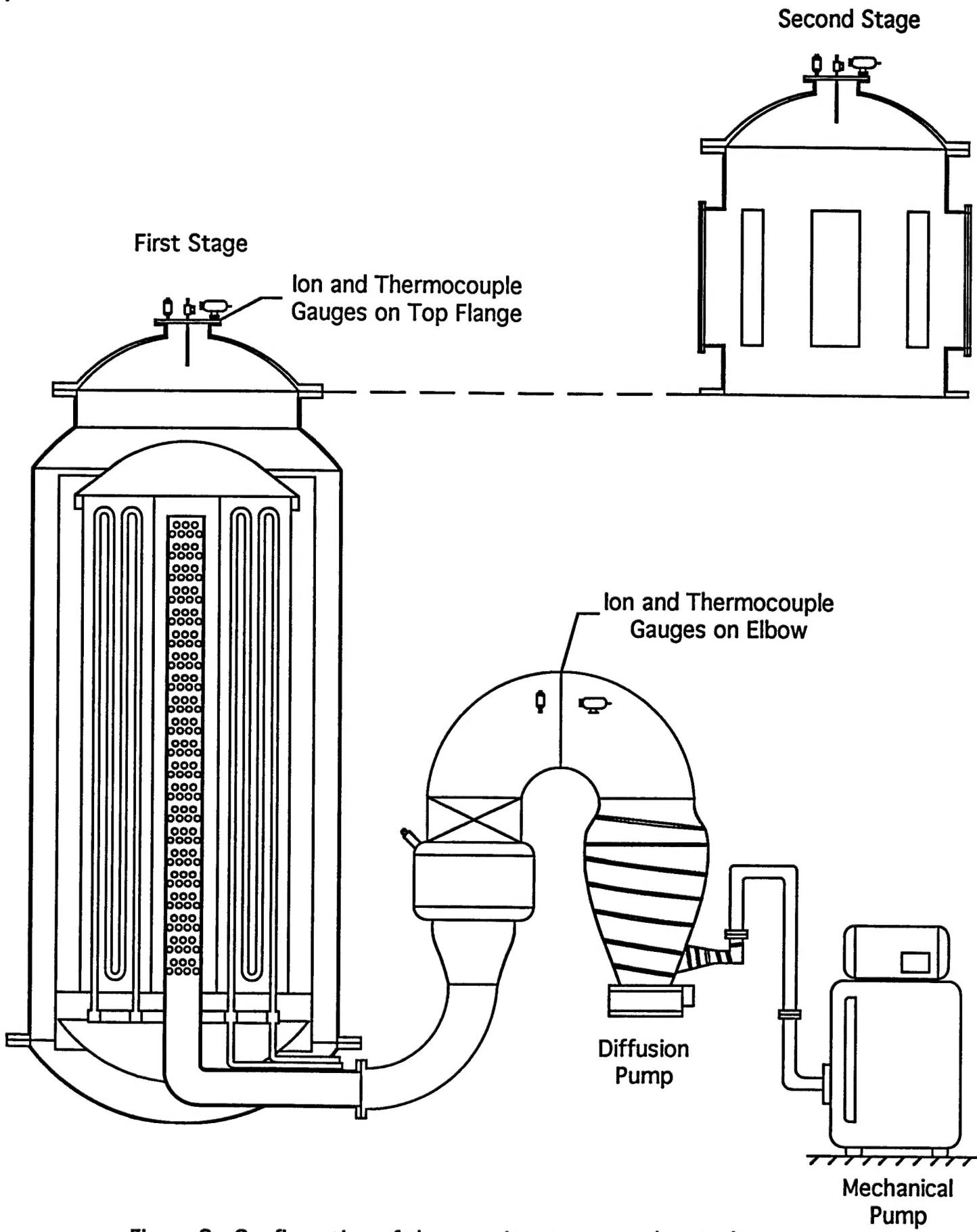


Figure 2: Configuration of the pumping system and stage layout.

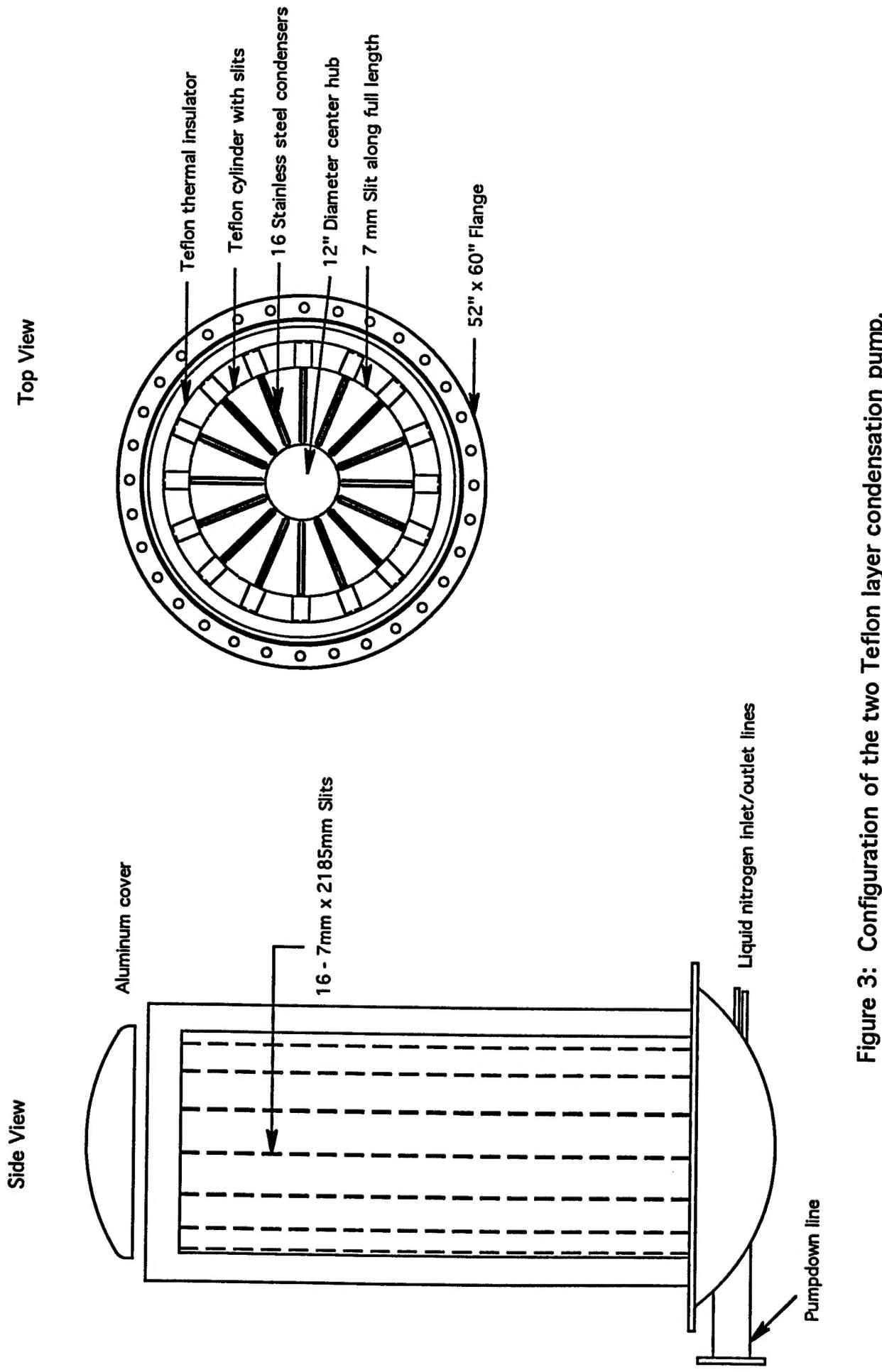
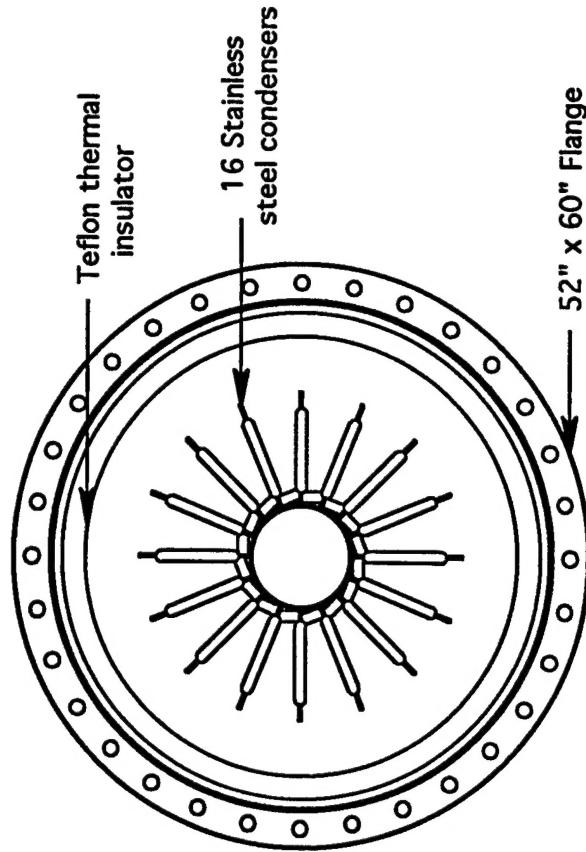


Figure 3: Configuration of the two Teflon layer condensation pump.

Top View



Side View

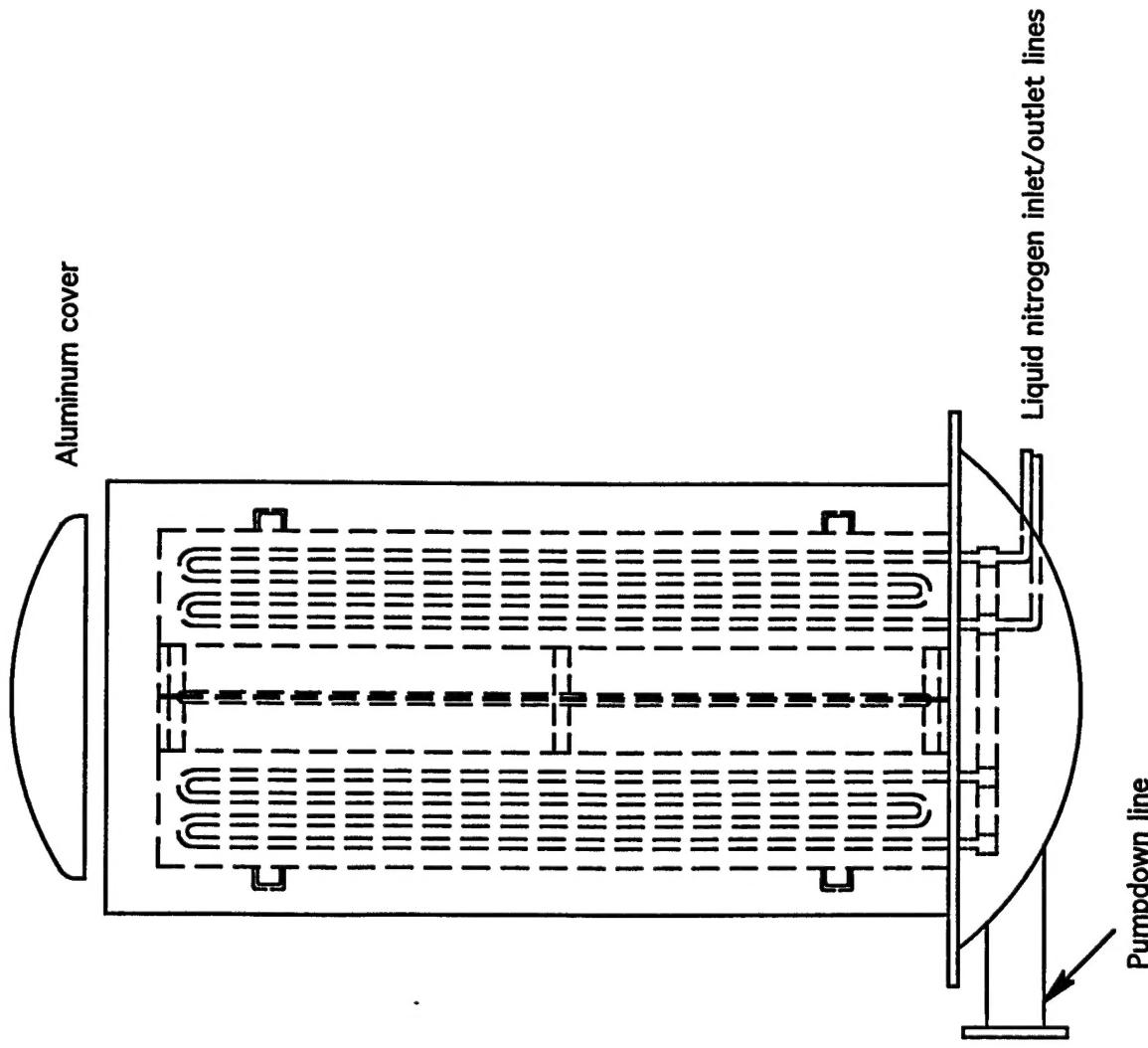


Figure 4: Configuration of the single Teflon layer condensation pump.

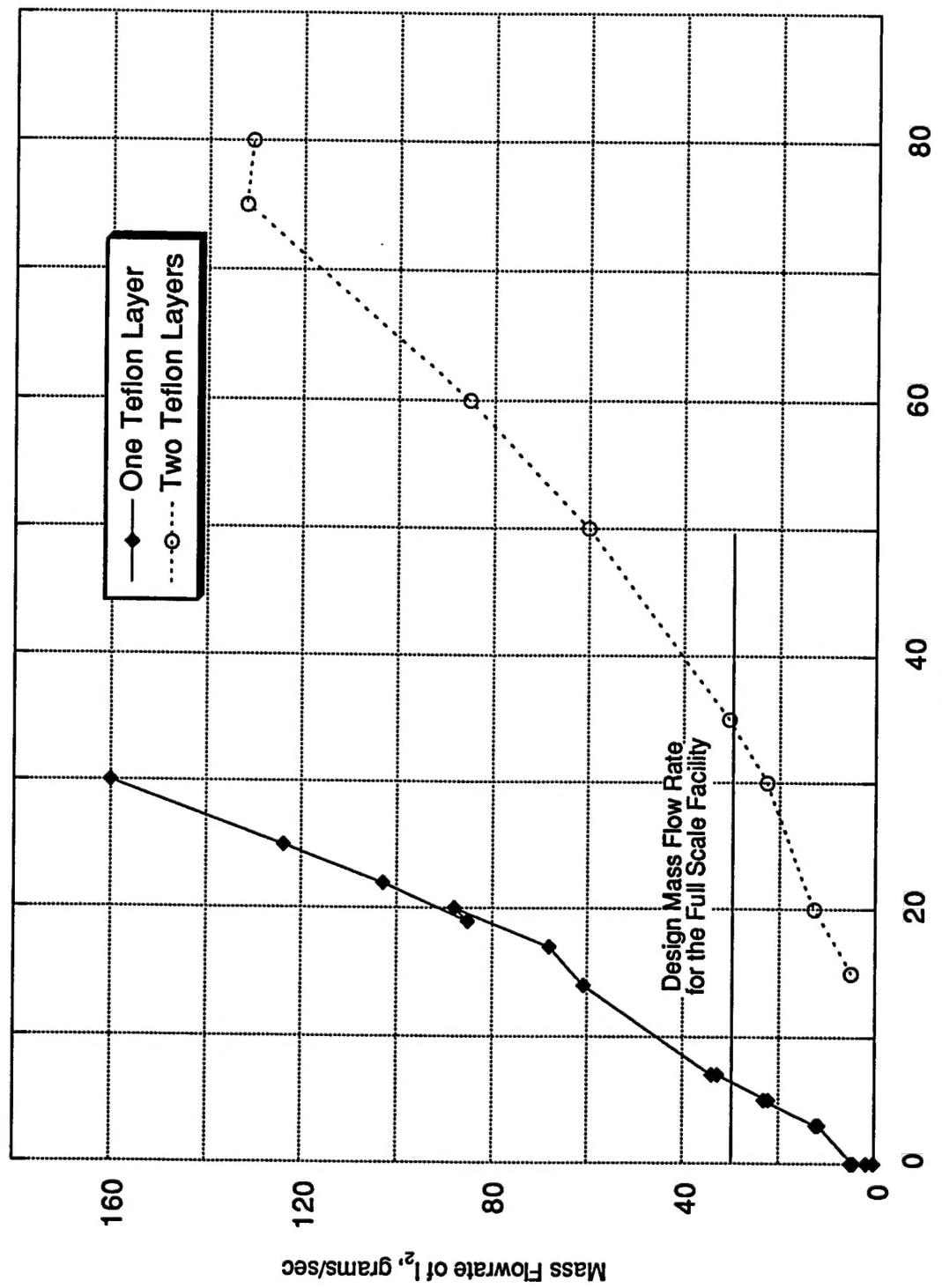


Figure 5: Simulation of the gas load requirements on the cryogenic panels.

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